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High-Cycle Fatigue Behavior of a Nicalon™ / Si-N-C Composite

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August 1999

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Abstract

Elevated temperature, high-cycle fatigue behavior of a woven SiC/Si-N-C ceramic matrix composite system was investigated at 910°C. High frequency (100Hz) fatigue tests were conducted in air on specimens machined from the composite system. A power-law type fatigue life relationship adequately characterized the high-cycle fatigue data generated in the study. Post failure fractographic and metallographic studies were performed to document the fatigue crack initiation regions and damage mechanisms in the composite system. Fatigue cracks initiated primarily from the corners of the specimens and propagated along the 90° fiber tows.

Introduction

Ceramic matrix composites (CMC's) are under development for potential use in aerospace propulsion applications, such as turbine rotors, combustor liners, ducting, and exhaust nozzle components (1). In these applications, CMC components will experience complex thermal and mechanical cyclic loads. Several recent studies have examined the behavior of this class of materials under cyclic tensile loading (2-6). In a few studies, significant internal heating during cyclic loading of fiber reinforced ceramics was reported (2-4). For $[0/90]_{13S}$ C/SiC and $[0]_{16}$ Nicalon^{TM}/CAS composites, the temperature rise measured during cyclic loading was found to be dependent on peak stress and test frequency (2). Frictional sliding between the fibers and the matrix was postulated as being responsible for the heat generation (2-4).

In the previous studies, test frequencies ranging from 1 to 100 Hz were employed to investigate the effects of fatigue loading history and microstructural damage on the magnitude of internal heating and interfacial shear stress of laminated CMC's (2-4). No study has reported the high frequency fatigue behavior of a CMC with a woven fiber architecture at elevated temperatures. The purpose of this study was to examine the feasibility of fatigue testing a woven fiber reinforced CMC at an elevated temperature under high frequency conditions in order to obtain life data and to characterize the damage mechanisms. Tests were conducted at an elevated temperature to approximate the steady-state operating condition of a hot section aerospace propulsion system.

Material, Specimens, and Test Procedure

The material studied in this investigation was a woven SiC fiber-reinforced Si-N-C matrix composite (SiC/Si-N-C) manufactured by Dow Corning under the trade name of Sylramic™ S200 using the polymer impregnation and pyrolysis (PIP) method (7). The Si-N-C matrix is reinforced by eight plies of ceramic grade Nicalon™ fabric (8 harness satin weave) in a cross-ply layup $[0/90]_{4S}$. Dow corning manufactured the composite system using a proprietary interface material. However, two commonly used interface materials within similar types of CMCs are C and BN (4,5,8). Fiber volume fraction for the investigated Sylramic™ S200 was approximately 45%. The density was $2.1 g/cm^3$ and the composite contained open porosity of about 5%. The specimen employed was 152 mm long, with a grip section width of 12.7 mm, a reduced gage section width of 10.2 mm, and a thickness of 3.0 mm (9). Specimens were machined from composite plates using diamond grinding.

Axial fatigue tests were performed at 910°C in air with a test frequency of 100 Hz. A sinusoidal load control waveform with an R-ratio (minimum load/maximum load) of 0.1 was used. A high frequency servohydraulic test machine with water-cooled wedge grips was employed for the testing. The load train alignment was maintained to less than 5% maximum bending strain within the specimen. Heating of specimens was performed with an inductively-heated SiC susceptor. More details regarding the specimen heating, gripping and load train alignment can be found in (9). Temperature of the specimen was both controlled and monitored with two R-type (Pt-Rh) beaded thermocouples. The thermocouple used to monitor the specimen temperature was within 1% of the nominal test temperature. During the course of fatigue tests neither an increase in the temperature of the specimen nor a decrease in the power supplied (required to maintain the specimen temperature) to the induction heater was noticed. Failure was defined as separation of the specimen into two pieces. If a specimen did not fail by 10 million cycles, then that test was considered a runout and was terminated.

Results

Fatigue Life Data

The tensile properties of the CMC at 910°C were estimated from the room temperature to 982°C data available in Ref. 7. At the test temperature, the Young’s modulus and proportional limit strength of the CMC are 94 GPa and 87 MPa, respectively. The ultimate tensile strength and strain to failure are 295 MPa and 0.68%, respectively. A plot of the maximum cyclic stress versus fatigue life is shown in Fig. 1. For each maximum stress condition, two specimens were tested. Scatter in the fatigue life at a given maximum stress was less than a factor of 2. The arrow indicates two runout tests. The peak stress (86 MPa) yielding the runout life was less than proportional limit strength of 87 MPa (7). In computing the fatigue life relationship shown in Fig. 1, data from the two runout tests were omitted. The fatigue life relationship for the SiC/Si-N-C composite at 910°C is as follows:

$$\sigma_{max} = 4400(N_f)^{-0.248}$$

where, σ_{max} is the maximum stress in MPa and N_f is the number of cycles to failure.

Damage mechanisms

Post failure fractographic and metallographic examination of the high-cycle fatigue (HCF) specimens was conducted to document the damage mechanisms. Crack initiation occurred primarily at the corners and near the edges of the specimens in the intra-tow regions of the CMC, which were often associated with porosity (Fig. 2). Occasionally, additional crack initiation sites were also observed in the matrix-rich regions along the edges of the specimens (Fig. 3).

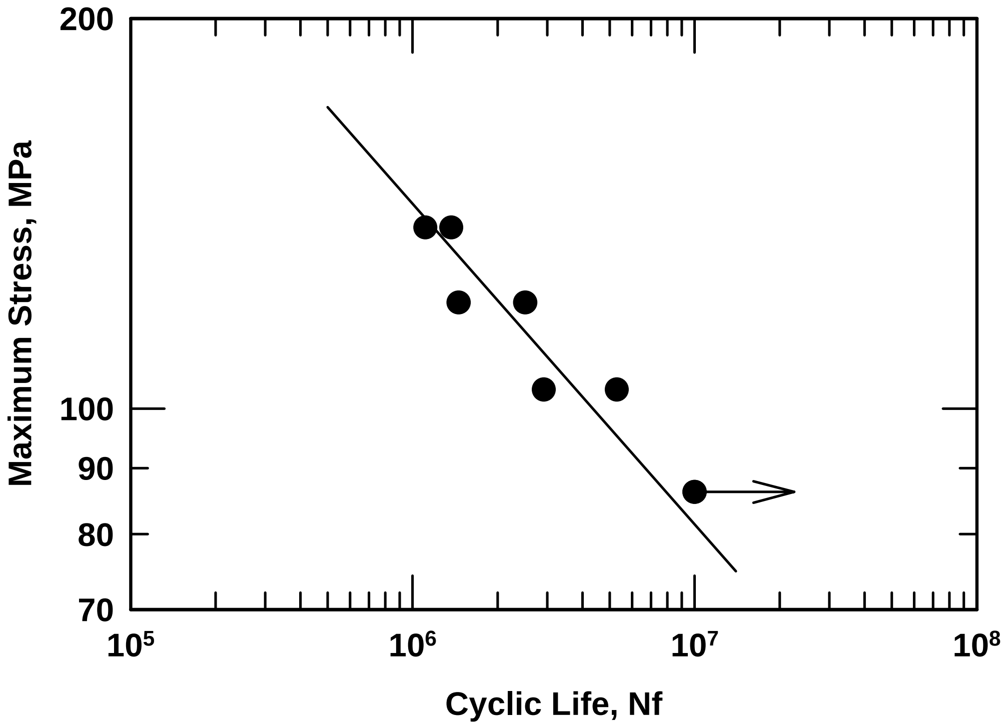


Figure 1. High-cycle fatigue data for Sylramic S200 composite at 910°C generated in air at a frequency of 100 Hz.

Cracks initiating at the corners and on the edges of both ends of the specimen propagated along the 90° tows, which were perpendicular to the loading direction. Broken fibers in a 90° tow bundle and a contiguous porous region adjacent to a 90° fiber-tow are clearly visible in Fig. 3. A few fatigued specimens were sectioned at various locations through the thickness and mounted in epoxy using a vacuum/pressure infiltration process to study the fracture topography variation along the width direction. A section near the corner of a specimen displayed nearly flat fracture surface with no significant fiber pullout confirming the earlier observation that crack initiation had indeed occurred near the corners of the specimen (Fig. 4a). Another section in the middle of the same specimen exhibited substantial fiber pullout, which is typically associated with an overload (Fig. 4b). Thus, the crack initiation and propagation mechanisms observed in the fracture surface were corroborated with the results from the metallographic examination. The fracture surfaces of the CMC did not indicate any significant degradation of either the matrix or the fibers due to environmental exposure. This observation suggests that the test temperature (910°C) and frequency (100 Hz) were not sufficient to precipitate any significant environmental damage mechanisms in the material.

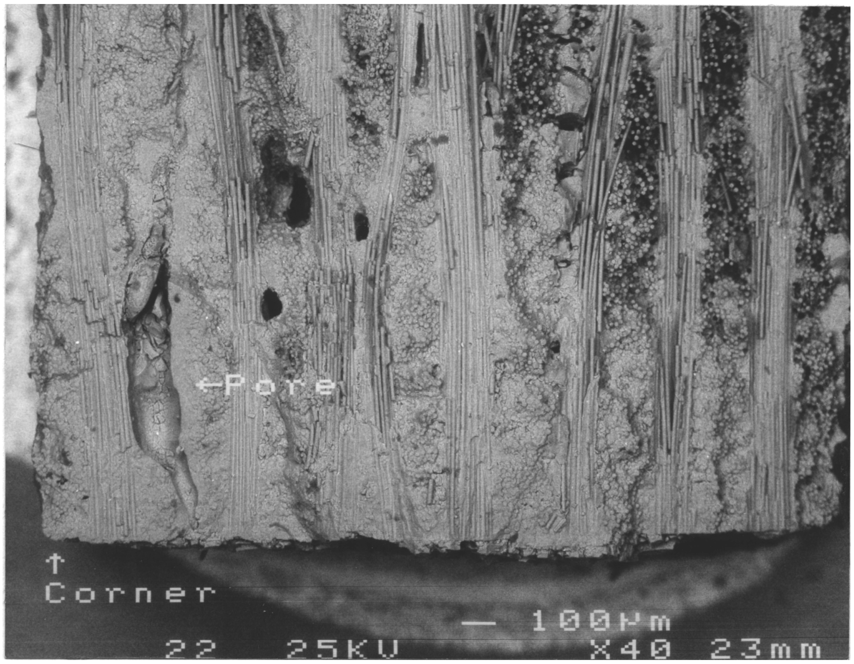


Figure 2. Fracture surface showing crack initiation from a corner and porosity in a specimen tested in HCF; σ_{max} = 138 MPa; and N_f = 1, 374, 600 Cycles.

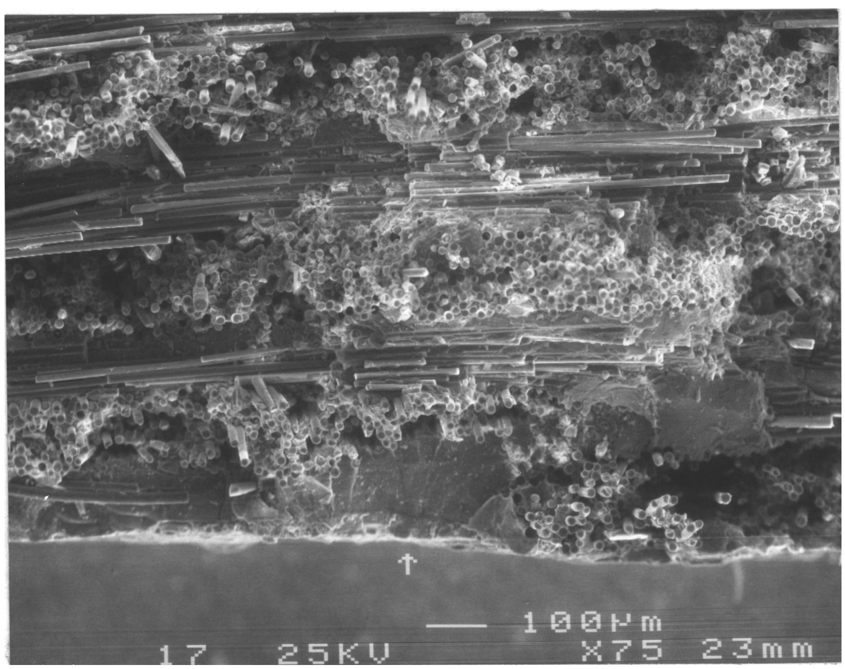
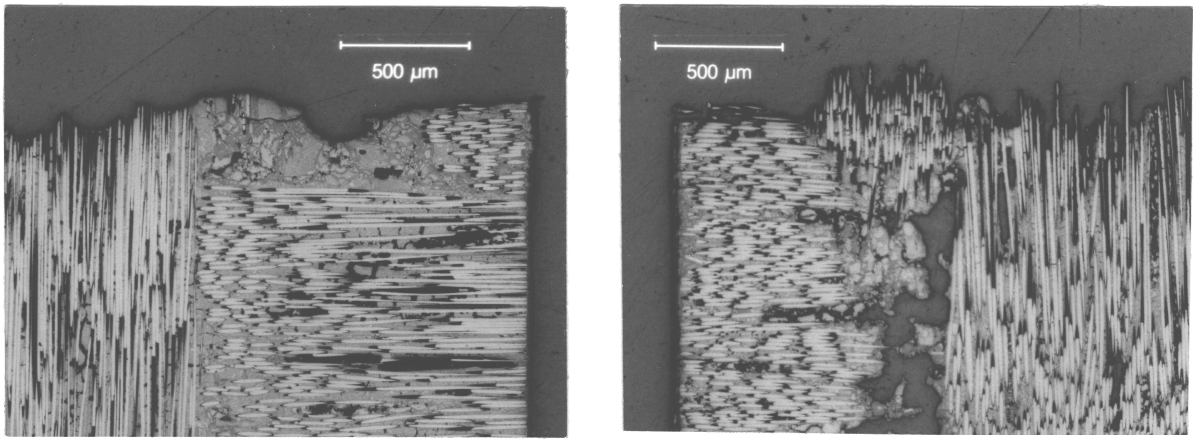


Figure 3. Fracture surface showing crack initiation in matrix-rich region at the edge of the specimen; σ_{max} = 103 MPa; and N_f = 2, 918, 900 Cycles.



(a) Flat fracture surface near a corner of the specimen (b) Fiber pull-out in the middle of the specimen

Figure 4. Variation of fracture surface topography in a HCF specimen;
σ_{max} = 138 MPa; and N_f = 1, 374, 600 Cycles.

Discussion

The HCF data of woven SiC fiber reinforced Si-N-C matrix composite at 910°C exhibited only a nominal amount of scatter and the material's fatigue life was characterized with a power-law type of life relationship similar to the stage II type of life relationship proposed by Talreja (10). Since the specimens used in the program were machined from two different plates, nominal scatter observed in the fatigue data and the associated deterministic nature of the HCF behavior of the CMC indicate that material and specimen fabrication processes were consistent.

As mentioned earlier, no significant degradation of either the fibers or the matrix was observed in the post failure examination of the CMC specimens. Verrilli et al. (8) reported erosion of fibers in a Nicalon™ fiber-reinforced SiC/SiC plain-woven composite manufactured by chemical vapor infiltration process and tested under tensile creep. The erosion of fibers was attributed to a "pesting" phenomenon exhibited by the SiC/SiC composite within the temperature range of 700 to 800°C. Lack of erosion of the Nicalon™ fibers within the SiC/Si-N-C system was likely due to the higher test temperature (910°C) used in the present study. In addition, differences in the matrix composition and composite processing techniques may have contributed to the integrity of the SiC/Si-N-C system.

Damage due to frictional sliding between the fibers and the matrix has been identified as a potential failure mechanism for some CMC's by Holmes (3). In the present study no such damage was observed in the SiC/Si-N-C composite system during microstructural examination. In addition, during the HCF testing of the SiC/Si-N-C composite at 910°C, neither an increase in the temperature of the specimen nor a reduction in the power consumption of the heating system was observed. The propensity for frictional sliding might have been reduced in SiC/Si-N-C composite system due to lowering of residual stresses within the woven fiber cloth and the matrix at the elevated testing temperature of 910°C. The residual stresses both in the fibers and the matrix at room temperature tend to be significantly higher than at 910°C because the latter is much closer to the processing temperature of the composite. The phenomenon of frictional heating was investigated by Holmes (3) in four different CMC's: $[0/90]_{13S}$ C/SiC, $[0]_{16}$ Nicalon^{TM}/CAS, $[0/90]_{4S}$ Nicalon^{TM}/SiC, and SiC/Si_3N_4. Under cyclic loading at room temperature, significant increase in the specimen temperature was reported by Holmes (3) only in the C/SiC and Nicalon^{TM}/CAS composite systems. Holmes (3) also reported that no significant rise in the temperature was observed for the Nicalon^{TM}/SiC and SiC/Si_3N_4 composite systems, which are similar to the SiC/Si-N-C system investigated in the present study. It is possible that the chemical compatibility of the constituents of the CMC's (fibers and matrices) and minimization of the differences in the thermal coefficients of expansion of the fibers and matrices, which in turn reduces the magnitudes of the residual stresses within these constituents, can mitigate the frictional heating effects in the CMC's.

Summary

High frequency fatigue tests were successfully conducted on a woven $[0/90]_{4S}$ SiC/Si-N-C composite system at 910°C. No evidence of frictional heating was observed during elevated temperature fatigue testing. The high-cycle fatigue behavior of the CMC was characterized by a power-law type fatigue life relationship and post failure fractographic and metallographic examinations were conducted to identify the damage mechanisms. During high-cycle fatigue, cracks initiated primarily at the corners of the specimens and propagated along the 90° fiber tows. No deterioration or erosion of fibers was observed during the microstructural examination of the failed specimens.

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